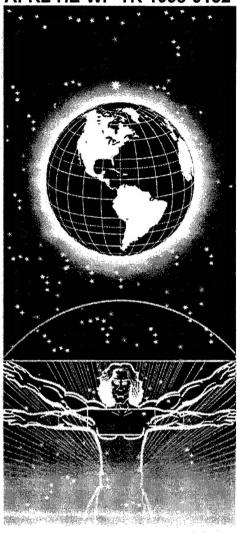
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UNITED STATES AIR FORCE RESEARCH LABORATORY

RISK ASSESSMENT FOR PHOSPHORUS TRIBROMIDE

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TECHNICAL REVIEW AND APPROVAL

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The animal use described in this study was conducted in accordance with the principles stated in the "Guide for the Care and Use of Laboratory Animals", National Research Council, 1996, and the Animal Welfare Act of 1966, as amended.

This report has been reviewed by the Office of Public Affairs (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

FOR THE DIRECTOR

STEPHEN R. CHANNEL, Maj, USAF, BSC Branch Chief, Operational Toxicology Branch Air Force Research Laboratory

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PREFACE

This is one of a series of technical reports describing a preliminary risk assessment for phosphorus tribromide tested at the Operational Toxicology Branch under the ManTech Geo-Centers Joint Venture contract. This document serves as a final report for the project conducted for phosphorus tribromide as a potential Halon replacement. The research described in this report began in October 1996 and was completed in March 1997 under Department of Defense Contract No. F41624-96-C-9010. Lt. Col Terry A. Childress, served as Contract Technical Monitor for the U.S. Air Force, Armstrong Laboratory. This study was sponsored by WL/MLBT.

The animal use described in these studies was conducted in accordance with the principles stated in the "Guide for the Care and Use of Laboratory Animals", National Research Council, 1996, and the Animal Welfare Act of 1966, as amended.

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ABBREVIATIONS

ACGIH American Conference of Governmental Industrial Hygienists

BTU/hr British thermal units per hour

°C Degrees centigrade
CaCO₃ Calcium carbonate
cc Cubic centimeter
cm² Square centimeter

cm²/s Square centimeter per second

cm³ Cubic centimeter

DoD Department of Defense

EC₅₀ Median effective concentration

F-344 Fischer 344 (rats)

ft Feet g Grams

g/cm³ Grams per cubic centimeter

H₂0 Water

HBr Hydrogen bromide H₃PO₃ Phosphonic acid

LC₅₀ Median lethal concentration

m³ Cubic meter

mg/m³ Milligrams per cubic meter

mg/L milligrams per liter

mL Milliliter

NIOSH National Institute for Occupational Safety and Health

NOAEL No-obserable-adverse-effect-level

NOEC No Effect Concentration
NZW New Zealand White (rabbits)

OSHA Occupational Safety and Health Administration

PBr₃ Phosphorus tribromide
PEL Permissable exposure limit

ppb Parts per billion
ppm Parts per million
RH Relative humidity
TLV Threshold limit value
TWA Time-weighted average

ug/cm²s Microgram per square centimeter seconds

ug/L Microgram per liter

uL Microliter
U.S. United States

SECTION I

HAZARD ASSESSMENT

Phosphorus tribromide (PBr₃) has effective fire fighting properties which make it a possible replacement for Halon 1301. The DoD requires the development of a toxicity profile for replacement candidates of Halons, which includes the results from acute and subchronic toxicity testing. Many potential replacements, including phosphorus tribromide, have not been thoroughly investigated to determine their toxicological properties. The purpose of this report is to summarize the toxicity studies conducted for phosphorus tribromide and to conduct a preliminary risk assessment for this compound. The risk assessment will help determine if phosphorus tribromide (PBr₃) can be used as a Halon replacement compound.

Tests in a 500,000 BTU/hr test burner demonstrated that PBr₃ is an effective fire extinguishant. The test burner was quenched by only 0.2 mL PBr₃. This volume is several thousand times less than the amounts required of other Halon replacements to suppress fires. New fire extinguishant systems utilizing PBr₃ would occupy less volume, weigh less and require less mechanics than current systems. Also, PBr₃ has no ozone depleting potential since it is rapidly hydrolyzed in the troposphere.

Phosphorus tribromide reacts with moisture in the air and on wet surfaces to produce phosphonic acid and hydrogen bromide gas (HBr) according to the following reaction:

$$PBr_3 + 3H_2O \rightarrow 3HBr + H_3PO_3$$

There is limited toxicity information available in the literature for phosphorus tribromide and hydrogen bromide. No acute toxicity information is available for phosphonic acid or hydrobromic acid, the aqueous form of HBr. The combined interaction of these compounds to cause potential health hazards is not known.

In a study of rats exposed to 1300 ppm HBr for 30 minutes, nose-breathing effects were compared to pseudo-mouth-breathing effects. Tissue injury in the nasal region of the respiratory tract was observed, including epithelial and submucosal necrosis. Pseudo-mouth-breathing exposure to HBr caused higher mortality rates and major tissue disruption in the trachea.

Observations of the trachea included necrosis of the epithelium, submucosa, glandular tissue and cartilage (Stavert *et al.*, 1991). In a separate study, the rat LC₅₀ for one hour was 2858 ppm while the mouse LC₅₀ for one hour was 814 ppm (RTECS, 1995a).

Human inhalation of 1300 to 2000 ppm HBr over a period of minutes was reported to be lethal (HSDB, 1995a). A short exposure to 35 ppm caused throat irritation (HSDB, 1995b), while exposure to 5 and 6 ppm for several minutes by 6 human volunteers also resulted in nose (6 out of 6) and throat irritation (1 out of 6) without eye discomfort (6 out of 6) (ACGIH, 1991a; HSDB, 1995c).

An accidental human exposure to PBr₃ and HBr was reported by Kraut and Lilis (1988). While mixing PBr₃, a female laboratory assistant was exposed to PBr₃ and HBr via splashing on the face, chest and hair, and by inhalation of

resulting vapors. She remained in the area of the exposure for five to ten minutes. Immediate effects noted were complaint of dry cough, light-headedness and slight congestion of the throat. Over the next two weeks, the subject experienced increasing shortness of breath. Chest x-rays revealed bilateral lobe infiltrates resulting in a diagnosis of chemical pneumonitis. She was allowed to return to work a few months later, though dyspnea on exertion persisted and chest x-ray findings had not yet completely resolved. Recovery was slowed by a number of relapses, apparently due to exposure to other respiratory irritants (Kraut and Lilis, 1988). No exposure assessment was conducted and attempts to get exposure information were not successful.

Results of genotoxicity testing of bacterial Salmonella strains indicated PBr₃ is not a mutagen. These results were consistent for frame shift and basepair substitution tester strains in both buffered and unbuffered solutions (ManTech, 1996).

No ecological dose-response levels were reported for HBr. Acute aquatic toxicity tests on fathead minnows ($Pimephales\ promelas$) and $Daphnia\ magna$ were recently conducted with Pbr₃. Fathead minnows (10 per group) were exposed to 0.0, 6.25, 12.5, 25.0, 50.0 or 100.0 mg/L PBr₃ in fresh water (88 mg/L hardness as CaCO₃) at 22 \pm 2 °C. The static tests were performed in replicate and the results were pooled. The 96-hour LC₅₀ value for fathead minnow was 71 mg/L (50 - 100 mg/L confidence limit); the no observed effect concentration (NOEC) was 25 mg/L (Aqua Survey, 1996a). These results indicate that an

isolated or intermittent exposure to a concentration of PBr₃ equal to 71 mg/L, is likely to cause death to 50 percent of fathead minnows, *Pimephales promelas*. A concentration equal to or less than 25 mg/L is not likely to have an adverse effect. Daphnia (10 per group) were exposed to 0.0, 6.25, 12.5, 25.0, 50.0 or 100.0 mg/L PBr₃ in fresh water (88 mg/L hardness as CaCO₃) at 20 ±2 °C. The static tests were performed in replicate and the results were pooled. The 48-hour EC ₅₀ value for *Daphnia magna* was 22.6 mg/L (18.2 - 27.9 mg/L confidence limit); the NOEC was 6.25 mg/L (Aqua Survey, 1996b). These results indicate that an isolated or intermittent exposure to a concentration of PBr₃ equal to 22.6 mg/L is likely to cause mortality/immobilization to 50 percent of the Cladoceran *Daphnia magna*, while a concentration equal to or less than 6.25 mg/L is unlikely to have an adverse effect.

Acute and subchronic PBr₃ studies were designed to determine the effects following single, high-concentration exposures which could occur in accidents, as well as repeated, low-concentration exposures which could occur on flight lines or during maintenance. Application of 10 or 50 μL neat PBr₃ to intact skin of an anesthetized NZW rabbit caused edema and necrosis of the treated skin within 10 minutes of dosing. Microscopic examination confirmed necrosis of the skin and underlying areas, including skeletal muscle of the subcutis. Application of 10 or 50 μL neat PBr₃ to intact skin of an anesthetized NZW rabbit for 30 seconds followed by a one minute water wash resulted in necrosis of the entire skin, but not the underlying skeletal muscle. An acute 4-hour nose-only

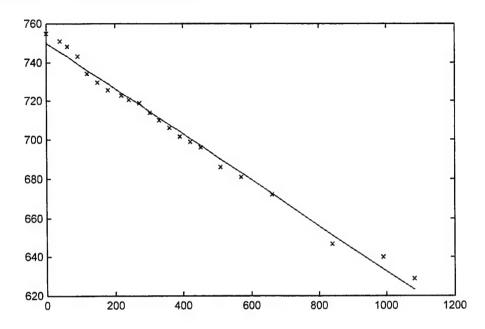
exposure of Fischer 344 rats to PBr₃ vapor resulted in mortality at 4.1 mg/L. At 1.5 mg/L, labored breathing, body weight loss, ulceration of anterior nares and rhinitis of the nasal passage were observed. Adverse effects were not observed in rats exposed for 4-hour to 0.4 mg/L. Male rats (5 per group) were exposed to PBr₃ vapor, 4 hours/day for 5 days, at 0, 0.06, 0.16 and 0.51 mg/L PBr₃. There were no signs of adverse effects at the low and mid-exposure levels. Rats in the 0.51 mg/L group had decreased body weights, gross lesions (reddened nares) and microscopic lesions (inflammation of mucosa and ulceration of epithelium in the nares). Rats (10 per sex per group) were exposed to PBr₃ vapor, 4 hours per day, 5 days per week, for 4 weeks at 0, 0.03, 0.1 and 0.3 mg/L. There were no signs of toxic stress, alterations in body weights or changes in organ weights in PBr₃ exposed animals. Minor serum chemistry and hematology effects were observed in the treated animals. Microscopic tissue findings were limited to rats of the 0.3 mg/L group and consisted of mild inflammation of the nasal passages. A concentration of 0.1 mg/L is the no-observable-adverse-effect level (NOAEL) in the 28-day inhalation study (Wolfe et al., 1997).

SECTION II

EXPOSURE ASSESSMENT

In order to calculate an inhalation exposure for PBr₃, the following information is needed: vapor pressure (2.25 Torr at 20 °C), molecular weight (270.7), density (2.8 g/cm³) and evaporation rate of the material as a function of temperature (7.6 μg/cm²s). The evaporation rate was based on an experiment conducted with a puddle of PBr₃ (4.755 g or 1.66 cc) with a surface area was 15.5 cm². The puddle was placed in a moderately ventilated room with a low relative humidity and the mass change was monitored over time at room temperature. Figure 1. shows the evaporation results, which fit a mass transfer (evaporation) rate of 7.6 micrograms per square centimeter per second. While this value may increase a few fold with more vigorous ventilation, it is still four orders of magnitude slower than evaporation into a vacuum. This material (PBr₃) is so dense and its vapor pressure so low that mass transfer becomes dominated by diffusion.

Figure 1. Milligrams of PBr_3 versus time in an open weigh boat with moderate ventilation at room temperature.



These numbers can be used to estimate the rate at which PBr₃ is released following a spill or accidental discharge. The density of PBr₃ is greater than aluminum; a spray, stream or aerosol will settle rapidly on the floor in the event of an accidental release.

After evaporation into the air, the material would react rapidly with atmospheric moisture to form HBr and H₃PO₃, according to the reaction:

$$PBr_3 + 3H_2O \rightarrow 3HBr + H_3PO_3$$
.

The hydrolysis rate for this reaction is 4 x 10⁻¹⁷cm²/s. This hydrolysis rate is based on the amount of water in air in molecules per cubic centimeter. At room temperature this quantity is expressed as the product of the relative humidity and the saturated vapor pressure of water (17.5 torr or 6.18 molecules per cubic centimeter). At 50% relative humidity (RH), the hydrolysis lifetime is 81 milliseconds. At 5% RH the hydrolysis lifetime increases to 810 milliseconds, while at 100% RH it decreases to 41 milliseconds.

The diffusion of PBr₃ vapor is very slow. The slow diffusion of the agent implies that its mixing will be primarily convective and subject to ventilation rates in the work area. Given the high hydrolosis rate even at low RH, PBr₃ would be expected to travel less than 1 cm from the liquid without reacting, under normal work ventilation conditions.

Based on the rapid reaction of liquid PBr₃ with water vapor, it is reasonable to assume that all of the released agent will be quickly converted to HBr and H₃PO₃. H₃PO₃ melts at 74 °C and is very soluble in water (300 grams

per 100 cc H₂O), so this material will not be airborne except as an aerosol. HBr, although also very soluble in water (221 grams per 100 cc water), is a gas at room temperature (20 °C). A conservative assumption for accidental release is, therefore, that all of the PBr₃ is rapidly converted to gaseous HBr and aerosol H₃PO₃, either on contact or by reaction in a zone less than a centimeter from the liquid.

Using this conservative model, the 10 cc PBr₃ cartridge (28.5 grams) that was tested in full scale engine nacelle tests should generate approximately 25.5 grams (0.315 moles or 7.05 liter atmospheres) of HBr. If all of the PBr₃ is converted to HBr and is uniformly distributed in a room 10x10x5 meters in size, the maximum concentration of HBr would be 14.1 ppm. The concentration is inversely proportional to the volume of the room; in a storage closet 2x2x2 meters in size, it would be 881 ppm. These concentrations are upper bounds since the gas will be immobilized as it freely dissolves in the moisture present on surfaces in the room. The rate of surface adsorption of HBr could be easily quantified by experiment if it is important for modeling smaller rooms. For direct comparison with the inhalation toxicology data, these concentrations can be expressed in mg/liter; 0.05mg/liter (51 mg/m³) for the small room and 2.96 mg/liter (2.961 mg/m³) for the closet release.

Although there is no typical hangar size, dimensions of the hangars at Wright-Patterson Air Force Base are approximately 200x50x8 ft or 61.5x46.2x14.8 meters. The total volume of such a hangar is 42,051 m³. If 25.5

grams of HBr is uniformly distributed in one of these hangars when it is empty, the maximum concentration would be 0.00061 mg/L (6.1x10-4mg/L) or 0.61 μg/L. This would be equal to 0.61 mg/m³ or 0.18 ppm or 180 ppb. This still is an upper bound because it assumes that all of the agent is sprayed over moist surfaces, through moist air and reacts 100% with the surface water and water vapor without any of it becoming dissolved in water.

SECTION III

RISK CHARACTERIZATION

For HBr exposure limits, National Institute for Occupational Safety and Health (NIOSH) set a ceiling limit of 10 mg/m³, Occupational Safety and Health Administration (OSHA) set a permissible exposure limit (PEL) of 10 mg/m³ as an 8-hr time weighted average (TWA) and American Conference of Governmental Industrial Hygienists (ACGIH) set a 10 mg/m³ threshold limit value (TLV) as a ceiling. The maximal hangar concentration of 0.61 mg/m³ would be an order of magnitude below the action level for these standards.

The NOAEL for the 28-day inhalation study was 0.1 mg/L for PBr₃. Since PBr₃ reacts to form HBr, the maximum possible concentration of HBr present at the NOAEL was 9.0x10⁻² mg/L or 90 mg/m³ (Wolfe *et al.*, 1997). Therefore, the maximal concentration of HBr in the hangar after an accidental discharge of 10 cc PBr₃ would be two orders of magnitude lower than the concentration of HBr at the NOAEL for PBr₃ in the 28-day study.

The issue then becomes one of using either the standards for HBr or the NOAEL for PBr₃ as exposure limits. Until a standard for PBr₃ is developed and accepted, the standard for HBr will most likely be used by industrial hygientists. Even after a standard is developed for PBr₃, exposure assessment will depend on the measurement of HBr since PBr₃ reacts so quickly with water vapor in the air. PBr₃ would not likely be used in anydrous or dessicated areas in real world situations.

Due to the density of the PBr₃ and slow diffusion rate, it will not travel far after accidental discharge, allowing time for personnel to exit the area. Due to its reactivity, it will convert to HBr which should achieve high concentrations only in small rooms. In the 10x10x5 meter room, the maximum concentration of HBr possible (51 mg/m³) is still lower (by almost one half) than the concentration of HBr present at the NOAEL for the 28-day PBr₃ study. There is still the issue of potential exposure to an individual at or very close to the point of release. Acid concentrations should to be measured or estimated for an accidental release scenario. The acid concentration could be determined for various heights and distances near the source in order to address this issue.

The primary hazard arises from direct contact with the skin. The use of this agent in occupied spaces is possible but may not be advisable in all cases. How PBr₃ is supplied, its placement in a system and industrial hygiene procedures can minimize the risks of its use.

SECTION IV

CONCLUSIONS

The reactivity of the agent with moisture and the solubility of the acid products in water make elevated airborne concentrations of PBr₃ and HBr extremely unlikely. The concentrations of HBr should be below the action level for exposure standards.

SECTION V

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